1910

A study of some assay problems

Walter Dobbins

Charles Weaver Traughber

Department: Materials Science and Engineering

Thesis

for the Degree of

Bachelor of Science.

1910.

A STUDY OF SOME ASSAY PROBLEMS.

By

Walter Dobbins. and C.W.Traughber.

Approved- Horace T. Mann

TABLE OF CONTENTS.

INTRODUCTION.

The object of this thesis is to work out some assay problems. Each problem is taken up independently, and may be considered as having no connection whatever with the others.

PROBLEM 1.

This problem has to do with the high litharge assay. Litharge is used in the fire assay for the purpose of slagging impurities, as well as to supply the lead necessary to collect the gold and silver values. Its prosence in a fusion keeps out of the lead button metals which cause much trouble in cupelling. Copper is the most frequently occurring of these troublesome metals, and all efforts should be made to keep it in the slag, and out of the lead. The presence of more than 2 or 3 % copper in the lead button injures cupellation.

To determine the efficiency of litharge as an absorbant of copper, we prepared a number of charges, all being similar except that the amount of litharge was varied. A mixture of equal weights, CuO and SiO_2, was the ore used.

Charge 1.

Soda ----------20 grams.
Litharge ----------30 grams.
Argols ----------2.5 grams.
Borax glass ---------10 grams.
Ore ----------0.5 assay ton.

Charge 2 --------Same as No 1, except 50 grams litharge.

" 3 -------- " " " " " 110 " "

" 4 -------- " " " " " 150 " "

" 5 -------- " " " " " 200 " "

" 6 -------- " " " " " 250 " "

" 7 -------- " " " " " 300 " "

" 8 -------- " " " " " 350 " "

Each charge was fused 45 minutes, at a temperature of approximately 1000 degrees Centigrade. All gave clean, liquid slags, free from lead shots. The slag and the button of each charge were weighed separately and analysied for copper. All copper determinations were made by the "Iodide Method". Table 1 and Curve 1 show the results obtained.

These results indicate that under the conditions named, the use of more than 250 or 300 grams of litharge, is not necessary. It is very difficult to lower the copper content in the lead button below 2 %.

Several cupellations of 2% copper buttons were made at a temperature of 700 degrees Centigrade, and no difficulty was experienced in feathering all of them. The cupels were free from cracks, and showed

but little discolorization from the copper oxide absorbed.

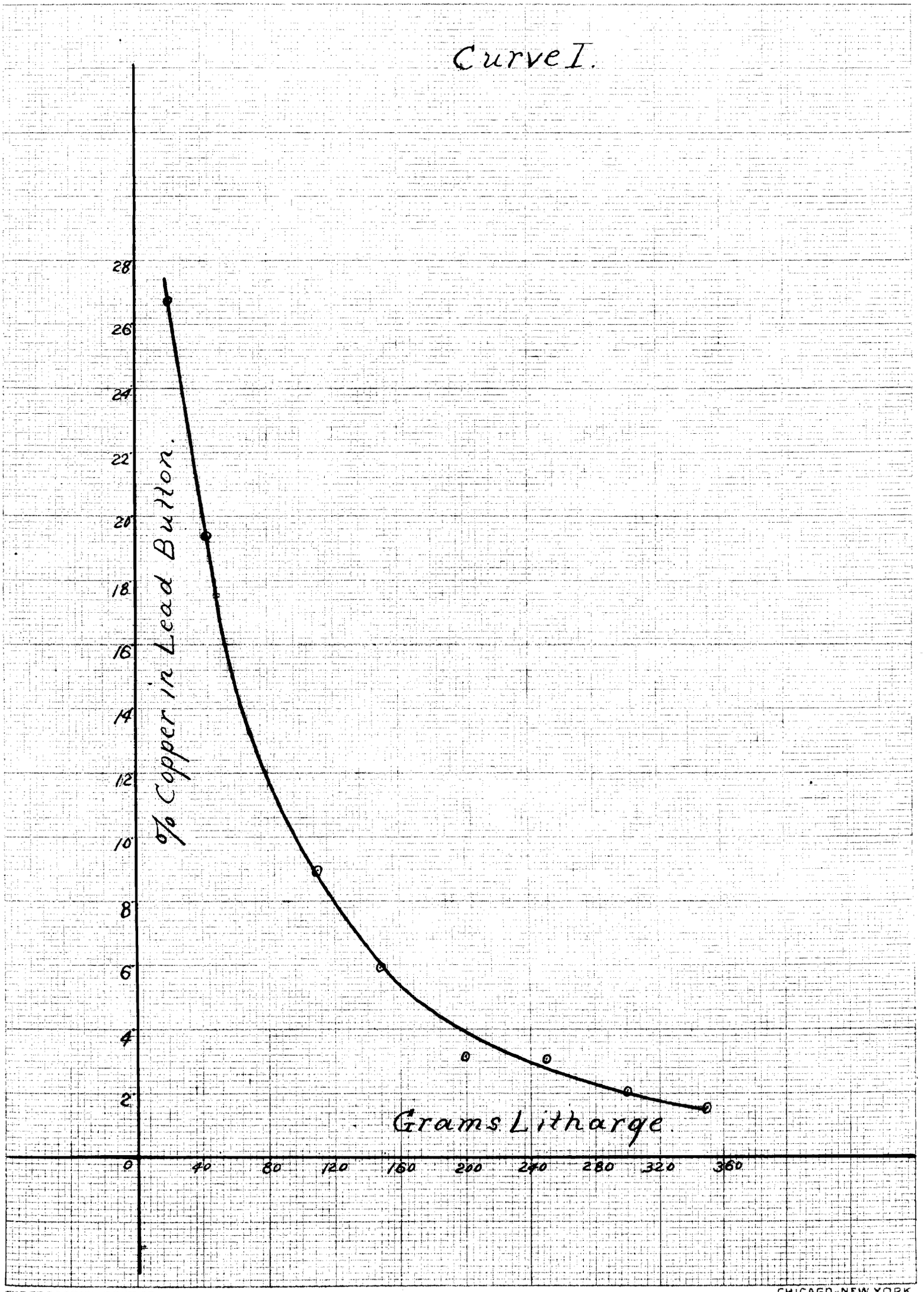
In summing up, we would say that no attempt should be made to lower the precentage of copper below 2% . It is shown that 2% copper does not injure cupellation, and hence it is unnecessary to use more than 250 or 300 grams of litharge in the above charge.

Table I.

Effect of Litharge

Temperature Constant (1000°C). Ore (½ Assay Ton).
Litharge Varying

Charge	Button in grams	% Copper in button	Grams Copper in button	Slag in grams	% Copper in Slag	Grams Copper in Slag
No 1.	18.1	26.66	4.82	19.	0.51	0.09
" 2.	19.2	19.56	3.75	50.	1.90	0.95
" 3.	19.0	8.87	1.68	111.	2.02	3.24
" 4.	18.5	5.96	1.10	150.	1.88	3.60
" 5.	18.9	3.18	0.60	205.	2.02	4.24
" 6.	18.2	3.01	0.50	234.	1.48	4.38
" 7.	19.1	2.02	0.38	309.	1.56	4.48
" 8.	19.3	1.48	0.26	365.	1.07	4.68


Curve I.
28
26
24
22
20
18
16
14
12
10
8
6
4
2
0
% Copper in Lead Button.
40
80
120
160
200
240
280
320
360
Grams Litharge.

PROBLEM 2.

The object of this experiment is to devise a scheme to eliminate the scorification of copper-lead buttons, when the percentage of copper is so high that it cannot be cupelled directly.

At present, the only method to prepare the button for cupellation is by slagging off the copper by scorification. The process of scorification requires from 20 to 30 minutes for completion, so that if a shorter method of getting rid of the copper could be substituted, much time would be saved.

Method:- After the lead button was obtained from the fusion of the ore, it was weighed and dropped into a bath of 100 grams of molten litharge. The charge was poured after 10 minutes heating, the button and slag weighed and assayed for copper.

We find that during this time, enough copper has been taken up by the litharge to fit the button for cupellation. Table 2 and Curve 2 show the adaptability of this process. The only difficulty in this operation was in preventing the attack and destruction of the crucibles by the litharge. This was overcome by giving the inside of the crucible a thick coating of iron oxide.

The results of this experiment show that this method of treating lead-copper buttons, could be substituted to advantage in place of scorification. Not only would from 10 to 20 minutes time be saved, but also the risk of getting too small a button be avoided.

Table II. (A)

Temperature-800°C – Time 10 minutes

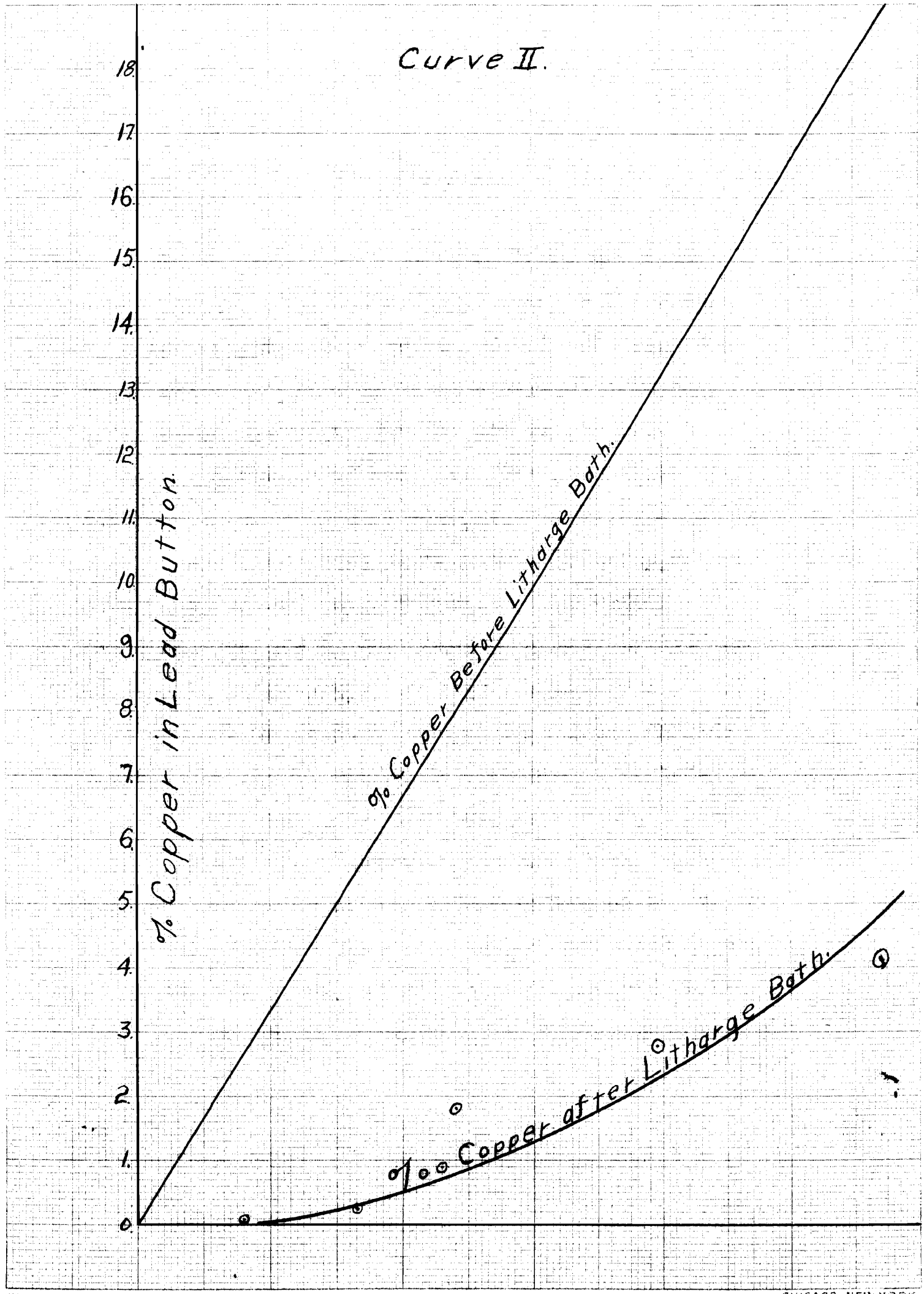
Litharge-100 grams.

Button	Button Wt. in grams	Grams after Pbo bath	Grams Loss in weight	% Cu. in button	% Cu. after Pbo bath	Grams Cu. in button before bath	Grams Cu. in button after bath
No 1	8.300	7.277	1.023	2.73	0.12	0.226	0.008
" 2	7.150	6.118	1.032	5.54	0.22	0.396	0.013
" 3	8.480	6.448	2.532	7.58	0.88	0.642	0.056
" 4	6.400	5.994	0.506	8.03	1.78	0.513	0.106
" 5	6.050	5.202	0.808	13.01	2.78	0.787	0.142
" 6	4.070	3.327	0.743	18.86	4.17	0.767	0.138

Table II (B).

Analysis of Litharge Slag

	Slag No. 1	Slag No. 2	Slag No. 3	Slag No. 4	Slag No. 5	Slag No. 6
% copper in slag	0.39	0.43	0.69	0.69	0.85	0.93
gms. copper in slag	0.39	0.43	0.69	0.69	0.85	0.93


Curve II.
% Copper in Lead Button.
% Copper Before Litharge Bath.
% Copper after Litharge Bath.
0.
1.
2.
3.
4.
5.
6.
7.
8.
9.
10.
11.
12.
13.
14.
15.
16.
17.
18.

PROBLEM 3.

The object of this problem is to try a new method of assaying heavy copper ores, and mattes. In order to get a lead button of suitable size for cupellation in presence of an excess of litharge, it is often necessary for the assayer to make a preliminary fusion of his ore. This new scheme is intended to do away with the preliminary, and at the same time to get a lead button free from copper and other impurities. Our idea was to thouroughly decompose the ore in the absence of lead or litharge , the result being simply a liquid slag, in which the gold and silver would be suspended . After the ore is fused, pour on the surface of the slag a mixture of argols and litharge or test lead, which would give a rain of lead to collect the gold and silver values. Not only could one obtain a lead button of the right size, but also one free from copper and other impurities, since they would all be oxidized and slagged before the lead was added. A number of different charges were

tried before we found one that was satisfactory. Each will be described in the order taken up.

A 31.87% copper matte, assaying 14.16 oz. gold and silver per ton, served as an ore. One half assay ton of the matte reduced 56 grams of the lead from an excess of litharge.

Charge 1.

Soda ---------- 20 grams.
Nitre ---------- 30 "
Borax glass -------15 "
Matte ----------0.5 assay ton.

The above charge was fused for 45 minutes at a high temperature. After the matte was apparently decomposed, we poured on the surface a mixture of 30 grams of litharge to 2.5 grams of argols (R.P.8). After 10 minutes further heating, the charge was poured. We obtained a 20 gram button, high in copper. Only a part of the matte was broken up. An explanation could not be made for the appearance of this matte, since the 20 grams of nitre in the charge, seemed ample to oxidize any sulphur present.

Charge 2.

This charge was the same as charge 1, except that the nitre was increased to 40 grams. The results were practically the same, the matte decomposition being little, if any, better than in Charge 1.

Charge 3.

Charge 3 was similar to charge 1 , the only difference being that in this case 30 grams of nitre was packed solidly in the bottom of the crucible, and the mixed ore and fluxes placed on top. No better success was obtained in breaking up the matte than in the two previous cases.

Charge 4.

Soda ----------20 grams
Nitre ----------25 "
Borax glass ----------10 "
Litharge ----------125 "
Silica ----------10 "
Matte ----------0.5 assay ton.

Charge 4 was fused for 35 minutes and then poured. This time the matte was broken up, but a 27 gram

lead-copper button was precipitated. The preliminary fusion which had been previously made, showed that 0.5 assay ton of the matte could throw down 56 grams of lead from an excess of litharge. Since our nitre had an oxidizing power of 3.9, it would seem that 14 grams should be sufficient to prevent a lead button coming down. In the above charge there was, theoretically, an excess of 11 grams of nitre above the amount necessary to keep a button from forming.

Charge 5.

Soda ----------20 grams
Nitre ----------40 "
Borax glass ----------10 "
Litharge ----------125 "
Silica ----------10 "
Matte ----------0.5 assay ton.

This charge formed well and there was no button present. The slag was homogeneous and free from matte. The above proportion of fluxes was used in all subsequent charges, since it was found satisfactory in decomposing the matte.

A series of 6 charges were prepared, using the same amount of matte and fluxes as in Charge 5. A mixture of 2.5 grams argols (R.F.2.5) and 30 grams of litharge, was poured on the surface of each fusion, as soon as it became quiet. The argols and litharge mixture was added in small necessitive amounts, so that the slags received from 1 to 6 washes of lead. Soft, malleable buttons, weighing from 20 to 21 grams were obtained. All were sufficiently free from copper to enable them to be cupelled at a temperature of 700 degrees Centigrade. The table below shows the results obtained.

1	lead	wash	10.68	oz.	Gold	and	Silver	per	ton.
2	"	"	9.60	"	"	"	"	"	"
3	"	"	11.84	"	"	"	"	"	"
4	"	"	8.56	"	"	"	"	"	"
5	"	"	11.28	"	"	"	"	"	"

A number of other assays were made, but in these cases test lead was substituted in place of argols and litharge. The results were lower than in the first case, the highest assaying but 4.67 oz. per ton.

In summing up this scheme, we may say that under our conditions, gave results from 4 to 5 ounces lower than the ordinary assay. It seems that the lead rain does not wet all the slag while descending to the bottom of the crucible. This was particularly so in the case of the test lead, which sank immediately in large globules upon being poured on the surface of the fusion. This assay might be successful if some means could be used to have the rain of lead come in contact with all parts of the slag. It certainly gives a button free from copper. The fact that the nitre did not break up the matte is another problem that remains unsolved. We made several determinations of the oxidizing power of the nitre, and found it to be from 3.8 to 4.0 grams of lead from litharge.

PROBLEM 4.

Problem 4 deals with the cupellation of a number of lead-copper buttons, of constant weight and copper percentage. We wish to find out whether or not there is a concentration of copper in the button as the cupellation proceeds. Five lead buttons were prepared, each containing 20 grams lead and 1 gram copper. This gives a button assaying 4.76% copper.

These five buttons were started cupelling at the same time and at the same temperature. The first button was withdrawn after 4 minutes cupellation, the second after 8 minutes, the third after 12 minutes etc., so that 5 buttons were obtained, representing successive periods of cupellation of from 4 to 20 minutes. Each of these fractional buttons was weighed and assayed for copper. Table 3 and Curve 3 show the data obtained in this experiment.

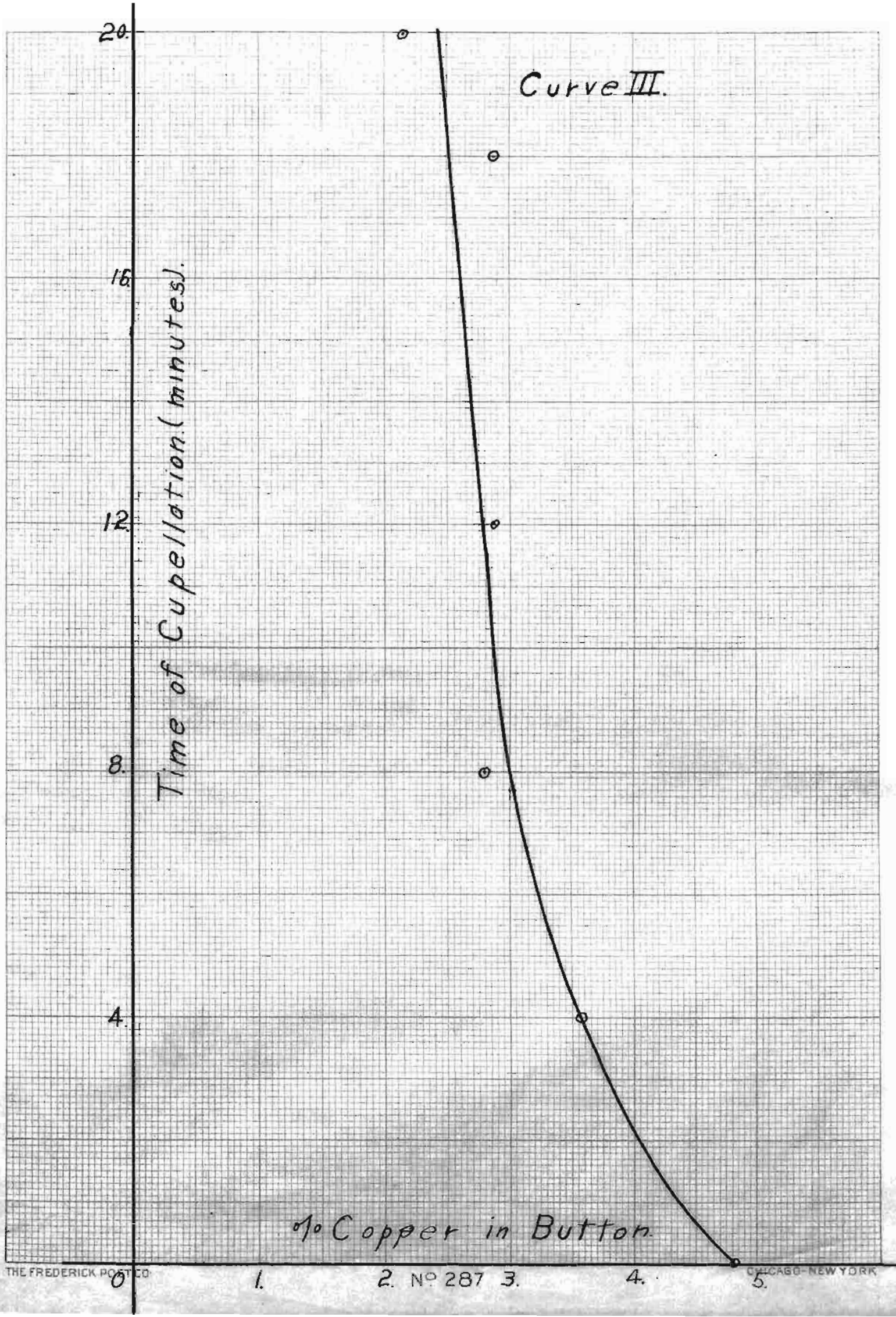
Curve 3 indicates that the percentage of copper decreases rapidly during the first four minutes, but that after this time the copper is absorbed at about the same rate as is the lead.

Table III.

Temperature (800°C). Weight Button 21 Gms.

Button 4.76% Copper

Button	Time of Cupellation Minutes	Weight Button grams	% Copper in Button	Copper in Button grams	Copper in Cupel grams
No. 1.	4	12.150	3.33	0.404	0.596
" 2.	8	10.550	2.80	0.295	0.705
" 3.	12	7.250	2.89	0.209	0.791
" 4.	16	3.670	2.86	0.104	0.896
" 5.	20	0.287	2.65	0.007	0.993


Curve III.
Time of Cupellation. (minutes).
20.
16.
12.
8.
4.
0
1.
2.
3.
4.
5.
% Copper in Button.
THE FREDERICK POST CO
No 287
CHICAGO-NEW YORK